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4-Methyl-*N*-(prop-2-ynyl)-*N*-(1*H*-pyrrol-2-ylmethyl)-benzenesulfonamide

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Key indicators

Single-crystal X-ray study
T = 145 K
Mean σ(C–C) = 0.003 Å
R factor = 0.067
wR factor = 0.117
Data-to-parameter ratio = 20.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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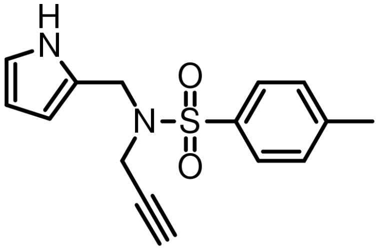
The crystal packing in the title compound, $C_{15}H_{16}N_2O_2S$, consists of an N—H$\cdots\pi_{\rm pyrrole}$, a C—H$\cdots\pi_{\rm pyrrole}$, a C—H$\cdots\pi_{\rm phenyl}$ and two C—H$\cdots$O interactions.

Comment

The title compound, (I), was prepared in an effort to extend the gold-catalysed synthesis of highly substituted phenols with well defined substitution patterns, from furans as starting materials to the corresponding pyrroles which should provide anilines (for background information, see Hashmi *et al.*, 2000). The title molecule (Fig. 1) has two intramolecular contacts which approach the van der Waals contact distance: H5*A*$\cdots$O1 2.43 Å and H6*B*$\cdots$O2 2.44 Å. The pyrrole group is planar within experimental uncertainty. The phenyl group shows a small deviation from planarity: atoms S and C15 deviate by 0.061 (3) and 0.081 (4) Å, respectively, in the same direction from the phenyl plane. These deviations from planarity may result from crystal-packing effects.



(I)

The crystal packing shows five intermolecular interactions [Fig. 2 and Table 1: C*g*(pyrrole) is the centroid of the pyrrole ring and C*g*(phenyl) is the centroid of the phenyl ring]. The alkyne group acts as a donor of a C—H$\cdots$O hydrogen bond with a rather short H$\cdots$O distance of 2.37 Å. The C1—H1 bond also is involved in a C—H$\cdots$O interaction, but with an H$\cdots$O distance of 2.45 Å. The pyrrole groups form a herring-bone pattern in the **b** direction. Neighboring pyrrole groups are connected by N—H$\cdots\pi$ interactions. The N—H donors do not point to the centroid of the acceptor pyrrole ring, but point closely to atom C2 of the acceptor ring. The intermolecular H01$\cdots$C2 distance of 2.44 Å is rather short. N—H$\cdots\pi_{\rm pyrrole}$ interactions have also been reported by Bennis & Gallagher (1998) and Gallagher & Moriarty (1999). The crystal structure of pyrrole (Goddard *et al.*, 1997) shows a herring-bone pattern of pyrrole groups connected by N—H$\cdots\pi$ interactions, similar to the structure of (I). The angle between neighboring pyrrole groups is 70.1° in the crystal structure of pyrrole, while a

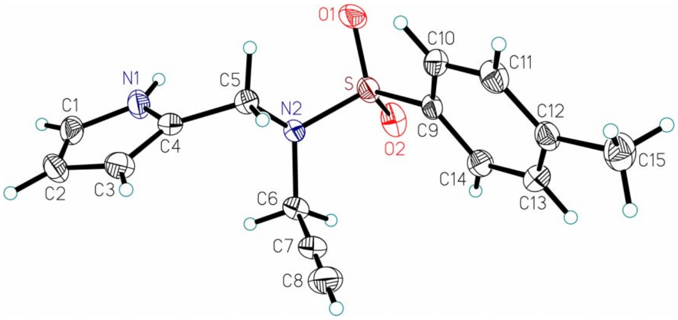


Figure 1
The structure of (I) with 50% probability displacement ellipsoids.

similar value of 69.4 (1)° is observed in (I). Intermolecular C5—H5*A*···$\pi_{\rm pyrrole}$ interactions further stabilize the crystal structure. The C5—H5*A* bond points closer to the C2—C3 bond than to the centroid of the acceptor pyrrole group. Intermolecular C15—H15*C*···$\pi_{\rm phenyl}$ interactions connect neighboring tosylate groups in the **a** direction.

Experimental

Propargylamine (2.20 ml, 32.1 mmol) was added to a solution of pyrrole-2-carboxaldehyde (3.07 g, 32.3 mmol) in dichloromethane containing $MgSO_4$ (10 g, 83 mmol). The reaction mixture was stirred for 20 h at ambient temperature, then the solvent was removed *in vacuo* to afford (prop-2-ynyl)(1*H*-pyrrol-2-ylmethylene)amine (4.17 g, 31.6 mmol) in 98% yield. This crude product was dissolved in dry methanol, sodium borohydride (1.43 g, 37.9 mmol) was added at 288 K and the reaction mixture was stirred for 1.5 h at room temperature. After addition of 100 ml water and extraction with dichloromethane, the organic phase was dried over $MgSO_4$ and the solvent partially removed *in vacuo*. The resulting solution was mixed with triethylamine (8.80 ml, 63.2 mmol) and 4-(*N*,*N*-dimethylamino)pyridine (39.0 mg, 319 μmol). At 273 K, toluene-4-sulfonyl chloride (12.0 g, 63.2 mmol) was slowly added and the reaction mixture was stirred at 273 K for 15 min and another 16 h at ambient temperature. After aqueous work-up, the organic phase was dried over $MgSO_4$ and the solvent removed *in vacuo*. Column chromatography (silica gel, hexane/acetone = 3:1) provided 3.50 g (38%) of (I). Single crystals were obtained by slow evaporation of a solution in ether at 293 K.

Crystal data

$C_{15}H_{16}N_2O_2S$
$M_r = 288.36$
Monoclinic, $P2_1/c$
$a = 15.926$ (4) Å
$b = 8.345$ (4) Å
$c = 10.955$ (3) Å
$\beta = 99.50$ (4)°
$V = 1436.0$ (8) Å^3
$Z = 4$
$D_x = 1.334$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 54 reflections
$\theta = 3$–23°
$\mu = 0.23$ mm^{-1}
$T = 145$ (2) K
Block, colorless
0.5 × 0.4 × 0.1 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: numerical (*SHELXTL*; Sheldrick, 1996)
$T_{\rm min} = 0.887$, $T_{\rm max} = 0.978$
23670 measured reflections
3749 independent reflections
2144 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.096$
$\theta_{\rm max} = 29.0°$
$h = -21 \rightarrow 21$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$
135 standard reflections frequency: 600 min intensity decay: none

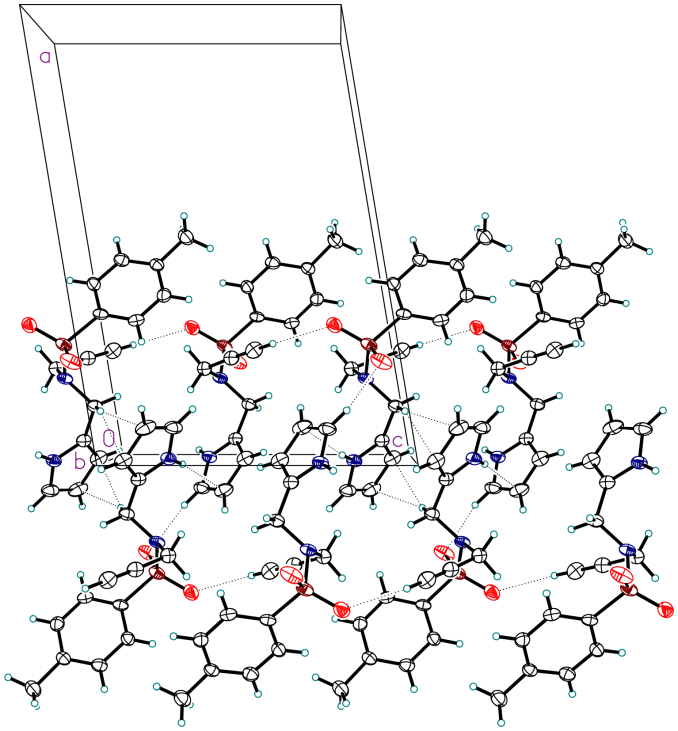


Figure 2
The crystal packing of (I), viewed down *b*.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.117$
$S = 1.04$
3749 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.33$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0049 (10)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1$^{\rm i}$	0.95	2.45	3.210 (4)	137
C8—H8···O2$^{\rm ii}$	0.95	2.37	3.240 (4)	152
C5—H5*A*···*Cg*(pyrrole)$^{\rm iii}$	0.99	2.61	3.395 (4)	136
C15—H15*C*···*Cg*(phenyl)$^{\rm iv}$	0.98	2.78	3.754 (4)	173
N1—H01···C2$^{\rm v}$	0.88	2.44	3.304 (4)	167

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $-x, 1-y, 2-z$; (iv) $1-x, y-\frac{1}{2}, \frac{5}{2}-z$; (v) $-x, \frac{1}{2}+y, \frac{3}{2}-z$.

The H atoms were taken from a difference Fourier synthesis. They were refined with fixed individual displacement parameters [$U({\rm H}) = 1.2U_{\rm eq}({\rm C_{non\text{-}methyl}})$, $U({\rm H}) = 1.5U_{\rm eq}({\rm C_{methyl}})$ and $U({\rm H}) = 1.2U_{\rm eq}({\rm N})$], using a riding model with fixed distances: H—N = 0.88 Å, H—C(secondary) = 0.99 Å, H—C(aromatic) = 0.95 Å, H—C(methyl) = 0.98 Å and H—C(alkynyl) = 0.95 Å.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL*97.

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